

**DEVELOPMENT OF NANOSTRUCTURED GAS  
SENSING MATERIAL AS AN ETHYLENE GAS  
DETECTOR**

**MAISARA AZAD BINTI MAT AKHIR**

**UNIVERSITI SAINS MALAYSIA**

**2016**

**DEVELOPMENT OF NANOSTRUCTURED GAS SENSING  
MATERIAL AS AN ETHYLENE GAS DETECTOR**

**by**

**MAISARA AZAD BINTI MAT AKHIR**

**Thesis submitted in fulfillment of the requirements  
for the degree of  
Master of Science**

**April 2016**

## ACKNOWLEDGEMENT

In the name of Allah, the Most Gracious and the Most Merciful. Alhamdulillah, all praises to Allah for the strengths and His blessing in completing this research and thesis successfully. Special appreciation to my main supervisor, Dr. Khairudin Mohamed, for his supervision, ideas and constant supports that always motivated me to complete this work. Also, to my co-supervisor, Dr. Sheikh Abbul Rezan, for invaluable supports, comments and suggestions throughout the project. Thank you from the bottom of my heart for your efforts which have contributed to the success of this project.

Not forgotten, to my collaborators at School of Chemical Science, USM, Dr. Lee Hooi Ling and Prof. Dr. A.S. Md. Abdul Haseeb from School of Mechanical Engineering, UM, I express my utmost gratitude for collaborating on this research and for all of the equipments and materials provided. This research would not have been completed without their willingness to work with me.

Special thanks to my colleagues, members of Nanofabrication and Functional Materials, USM, the demonstrators from School of Chemical Science USM and UM for their assistance and guidance. I want to express my gratitude to all staffs at School of Mechanical Engineering and School of Materials Engineering, USM for their cooperation and friendly atmosphere.

I also would like to thank Universiti Malaysia Perlis and Kementerian Pendidikan Malaysia for the financial support during this research. Last but not least, my deepest gratitude goes to my beloved parents, family members and friends for their endless love, prayers and encouragement. To those who indirectly contributed to this research, your kindness means a lot to me. Thank you very much.

## TABLE OF CONTENTS

Acknowledgement.....	ii
Table of Contents.....	iii
List of Tables .....	vii
List of Figures .....	x
List of Abbreviations .....	xxi
List of Symbols.....	xxii
Abstrak.....	xxiii
Abstract.....	xxv
CHAPTER 1 – INTRODUCTION	
1.1 Background of Study .....	1
1.2 Problem Statement.....	4
1.3 Objectives of The Research.....	5
1.4 Thesis Outlines.....	6
CHAPTER 2 – LITERATURE REVIEW	
2.1 Introduction.....	8
2.2 Gas Sensor and Gas Sensing Materials.....	8
2.2.1 Gas Sensor Characteristics.....	8
2.2.2 Type of Gas Sensors.....	11
2.2.3 Metal Oxide Semiconductor Gas Sensor.....	17

2.2.3.1	Fundamental Aspect of Metal Oxide Semiconductor Gas Sensors.....	18
2.2.3.2	Fabrication of Nanostructured Materials Based Gas Sensors.....	25
2.2.4	Ethylene Gas Sensing Materials.....	29
2.3	Tin Oxide, SnO <sub>2</sub> .....	31
2.3.1	Basic Properties of Bulk SnO <sub>2</sub> .....	32
2.3.2	Properties of SnO <sub>2</sub> Nanostructures and Its Gas Sensing Properties.....	32
2.3.3	Synthesis Methods of Tin Oxide Nanostructures.....	33
2.3.3.1	Chemical Vapor Deposition, CVD.....	35
2.3.3.2	Hydrothermal .....	42
2.4	Sensing Performance of SnO <sub>2</sub> Nanostructures.....	47
2.4.1	Reducing Gas .....	47
2.4.2	Oxidizing Gas .....	50

## CHAPTER 3 – METHODOLOGY

3.1	Introduction.....	54
3.2	Experimental Methodology .....	55
3.4	Design of Experiment, DOE .....	56
3.3	Synthesis Methods.....	59
3.3.1	Chemical Vapor Deposition (CVD) .....	59
3.3.1.1	Substrate Preparation .....	62
3.3.1.2	Growth Process.....	63
3.3.2	Hydrothermal Methods.....	65

3.3.2.1	Synthesis Process.....	66
3.3.2.2	Calculation & Substance Preparation.....	67
3.5	Characterization.....	70
3.5.1	Scanning Electron Microscope / Energy Dispersive Spectroscopy, SEM / EDS.....	71
3.5.2	X-Ray Diffraction, XRD.....	72
3.5.3	Ultraviolet-Visible Spectroscopy, UV-Vis.....	75
3.6	Gas Sensing Performance Characterization .....	77
3.6.1	Gas Sensing Measurement System .....	78
3.6.2	Gas Sensing Element Fabrication.....	80
3.6.3	Gas Sensing Test.....	82

## CHAPTER 4 – RESULT AND DISCUSSION

4.1	Introduction.....	84
4.2	Chemical Vapor Deposition .....	84
4.2.1	DOE Analysis of NWs Synthesis.....	87
4.2.2	Analysis of Variance (ANOVA) .....	88
4.2.3	Perturbation Analysis .....	91
4.2.4	Effect of CVD Temperature .....	93
4.2.5	Effect of Deposition Time.....	101
4.2.6	Effect of Argon Flow rate.....	105
4.2.7	Effect of Oxygen Flow rate.....	109
4.2.8	Optimization of Response by Cube Plot and Summary.....	116
4.3	Hydrothermal .....	117
4.3.1	DOE Analysis of NSs Synthesis by Hydrothermal .....	119

4.3.2	Analysis of Variance (ANOVA) .....	121
4.3.3	Perturbation Analysis .....	123
4.3.4	Effect of Precursor ( $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ ) Concentration.....	124
4.3.5	Effect of Temperature.....	131
4.3.6	Effect of Treatment Time.....	136
4.3.7	Effect of Pd Dopant.....	141
4.3.8	Optimization of Response by Cube Plot and Summary.....	146
4.4	Characterization of Gas Sensing Properties .....	147
4.4.1	Effect of Sintering Process to the Size of the Deposited Nanostructures.....	149
4.4.2	Gas Sensing Characteristic of $\text{SnO}_2$ NWs <i>via</i> CVD Route .....	153
4.4.3	Gas Sensing Characteristic of $\text{SnO}_2$ NSs <i>via</i> Hydrothermal .....	162
4.4.3.1	Crystal Size Effect.....	162
4.4.3.2	Effect of Pd Dopant.....	172
4.4.4	Comparison of Ethylene Gas Sensors's Performance <i>via</i> CVD and Hydrothermal Methods.....	180
CHAPTER 5 – CONCLUSIONS.....		186
REFERENCES.....		190
APPENDICES		
LIST OF PUBLICATIONS		

## LIST OF TABLES

		<b>Page</b>
Table 2.1	Response behavior of semiconducting gas sensor	10
Table 2.2	The advantages and limitations of different types of gas sensor	16
Table 2.3	Comparison of ethylene sensing materials	30
Table 2.4	Properties of ideal rutile structure of SnO <sub>2</sub> (Batzill and Diebold, 2005; Handbook of Chemistry and Physics, 2015)	32
Table 2.5	The literature summary of different synthesis techniques of SnO <sub>2</sub>	34
Table 2.6	Summary of SnO <sub>2</sub> NSs using CVD	41
Table 2.7	The literature summary of hydrothermal synthesis conditions	43
Table 2.8	The summary of gas sensing performance of different reducing gases	50
Table 2.9	The summary of gas sensing performance towards oxidizing gases	53
Table 3.1	The number of required experiments for CVD. (This table taken from Design Expert 6.0.8. software)	57
Table 3.2	The number of required experiments for hydrothermal (Generated by Design Expert 6.0.8. software)	58
Table 3.3	Materials and equipment for CVD synthesis	59
Table 3.4	Manipulated parameters for CVD synthesis	64



Table 3.5	Constant parameters for CVD synthesis	64
Table 3.6	Material and equipment for hydrothermal synthesis	65
Table 3.7	Variables and parameters for hydrothermal synthesis	67
Table 3.8	Molecular mass of the hydrothermal materials	68
Table 3.9	The main components in gas sensing measurement system in UM	79
Table 3.10	List of materials for sensor ink	81
Table 3.11	The Dilution of Ethylene Gas	83
Table 4.1	Experimental results of the diameter of the NWs via CVD using two level factorial design with 4 variables	88
Table 4.2	The summary of ANOVA for the diameter of the NWs and the deviation of Sn/O ratio	90
Table 4.3	Sn/O atomic ratio of Run 15 and Run 16	100
Table 4.4	Sn/O atomic ratio of NWs in Run 9 and Run 13	108
Table 4.5	Sn/O atomic ratio of Run 8 and Run 16	112
Table 4.6	Experimental results of the crystal size of the NSs via hydrothermal method using two level factorial design for 4 variables	120
Table 4.7	The summary of ANOVA for the crystal size	121
Table 4.8	Sn/O atomic ratio of NSs in Run 6 and Run 8	133
Table 4.9	Sample identification and descriptions	148
Table 4.10	The sensitivity of the CVD and hydrothermal samples at different operating temperature	153

Table 4.11	Response and recovery time of SnO <sub>2</sub> NWs towards 1000 ppm concentration of ethylene	155
Table 4.12	The sensitivity of the hydrothermal samples at different operating temperatures	164
Table 4.13	Response and recovery time of SnO <sub>2</sub> NWs and NSs towards 1000 ppm concentration of ethylene	175
Table 4.14	The sensitivity values of H11 and H16 at various ethylene gas concentrations	176
Table 4.15	Gas sensing properties of sensing materials obtained from both CVD and hydrothermal route at 1000 ppm concentration of ethylene gas	182
Table 4.16	Synthesis parameters for CVD gas sensors	182
Table 4.17	Synthesis parameters for hydrothermal gas sensors	182

## LIST OF FIGURES

		Page
Figure 1.1	Ethylene gas that was emitted by banana during the ripening process (Xiao <i>et al.</i> , 2013)	3
Figure 2.1	Types of gas sensor	11
Figure 2.2	Schematic capacitive gas sensor (Wagner, 2013)	12
Figure 2.3	IR-source gas sensors (a) based on the basic absorption spectrometry and (b) with reference filter/detector (Liu <i>et al.</i> , 2012)	14
Figure 2.4	Surface acoustic wave device operation (Wang <i>et al.</i> , 2009)	15
Figure 2.5	Schematic diagram of sensing process. (a) Receptor, (b) transducer and (c) resistance change. D: particle size, X: neck size, L = thickness of the space charge carrier (Park, 2010)	19
Figure 2.6	Receptor function of undoped SnO <sub>2</sub> and Pd doped SnO <sub>2</sub> (Park, 2010)	20
Figure 2.7	Schematic diagram of n-type semiconductor, SnO <sub>2</sub> . On the left: depletion layer formed in the surface in air, with the energy band bent up (high potential barrier); on the right: the reaction of oxygen with the reducing gas reduced the potential barrier (Park, 2010)	21
Figure 2.8	The sensitivity of SnO <sub>2</sub> toward H <sub>2</sub> and CO gases (Yamazoe, 1991)	23
Figure 2.9	Schematic diagram on the model of the effect of the crystallite size on the sensitivity of gas sensor (a) D >> 2Ls (b) D ≥ 2Ls (c) D < 2Ls (Rothschild, 2004)	24

Figure 2.10	Schematic showing the contribution of different components in a granular (Aswal, 2007)	25
Figure 2.11	Schematic of sensing material arrangement (a) Single NSs (b) aligned NSs (c) random distributed NSs (Arafat, 2012)	26
Figure 2.12	SEM images showing the steps of the in-situ lift-out fabrication procedure in the FIB/SEM system. (a) An intermediate ZnO nanorod on Si substrate, next to the FIB needle, (b) an intermediate ZnO nanorod – picked-up by the needle; (c) a single ZnO nanorod selected for sensor fabrication. Inset shows nanosensor substratetemplate (glass with Al contacts as contact electrodes); (d) a square hole cut on the glass; (e) place the ZnO nanorod over the hole; (f) single nanorod welded to both electrode /external connections as final sensor (Lupan <i>et al.</i> , 2008)	27
Figure 2.13	Random arrangement NSs (a) Film (Baik <i>et al.</i> , 2000) (b) Tube (Chen <i>et al.</i> , 2013)	28
Figure 2.14	SnO <sub>2</sub> cassiterite structure (Drake, 2007)	31
Figure 2.15	Outline of CVD technique process (Jones and Hitchman, 2009)	36
Figure 2.16	SEM images of (a) SnO <sub>2</sub> nanorods and a single nanorod of diameter 50–60 nm (inset) grown at 750°C temperature. (b) SnO <sub>2</sub> NWs at 850°C (c) SnO <sub>2</sub> nanobelt produce at 950°C (d) SnO <sub>2</sub> nanobelt at temperature 1000°C (Mondal <i>et al.</i> , 2010)	37
Figure 2.17	Growth process of (a) NWs (b) branched NWs and (c) nanobelts (Mondal <i>et al.</i> , 2010)	38

Figure 2.18	SEM images of SnO <sub>2</sub> thin films grown at temperature 420 °C for (a) 5 minutes, (b) 10 minutes, (c) 20 minutes, and (d) 30 minutes (Jeong, 2015)	40
Figure 2.19	Sequence of event during CVD (Pierson, 1992)	40
Figure 2.20	Schematic diagram of hydrothermal process (Xu and Sun, 2011)	42
Figure 2.21	FESEM images of SnO <sub>2</sub> nanorods at temperature (a) 120 °C (b) 140 °C (c) 200 °C and (d) 230 °C (Khuc <i>et al.</i> , 2010)	44
Figure 2.22	SEM image of SnO <sub>2</sub> nanoparticles at (a) 6 hours, (b) 12 hours, (c) 18 hours and (d) 24 hours (Zhang <i>et al.</i> , 2014)	45
Figure 2.23	Schematic of proposed formation process of flower-like SnO <sub>2</sub> assembled by nanorods (Zhang <i>et al.</i> , 2014)	46
Figure 2.24	XRD pattern of SnO <sub>2</sub> nanoparticles with pH level (a) pH 0.2, (b) pH 2, (c) pH 5 and (d) pH 9 at 130 °C for 6 hours (Kim <i>et al.</i> , 2009)	46
Figure 2.25	Schematic diagram of oxygen activity over (a) pure SnO <sub>2</sub> , (b) WO <sub>3</sub> /SnO <sub>2</sub> (nanoclusters) (c) WO <sub>3</sub> / SnO <sub>2</sub> (continuous layer) (Sharma <i>et al.</i> , 2012)	52
Figure 3.1	Experimental flow chart	55
Figure 3.2	The selected range of values of parameters and the desired responses for CVD methods. (This figure taken from Design Expert 6.0.8. software)	57

Figure 3.3	The selected range of value for each of parameters and the desired responses for hydrothermal methods (Generated by Design Expert 6.0.8. software)	58
Figure 3.4	Setup for CVD - Horizontal vacuum furnace	61
Figure 3.5	Schematic diagram of thermal evaporation process	63
Figure 3.6	The coating process of silicon substrate using thermal evaporator method	63
Figure 3.7	The schematic diagram of CVD process (bottom) for synthesis of SnO <sub>2</sub> NWs	65
Figure 3.8	Hydrothermal process flow chart	67
Figure 3.9	Schematic diagram of the UV-Vis	75
Figure 3.10	Process for fabrication gas sensing element and gas sensing test	78
Figure 3.11	The schematic diagram of the gas sensing measurement system. This system was connected to a computer via the specific interface board. Data were collected in the form of resistance and temperature in real-time	79
Figure 3.12	Alumina substrates with printed gold electrodes	81
Figure 4.1	$\Delta G$ versus temperature for both SnO and SnO <sub>2</sub> reactions	86
Figure 4.2	Graph predicted vs actual values of SnO <sub>2</sub> NWs diameter	91
Figure 4.3	(a) The perturbation plot of SnO <sub>2</sub> NWs diameter and (b) Contribution percentage and interaction of operating parameters in response to NWs's diameter	92

Figure 4.4	The effect of CVD temperature at (a) 850 °C (b) 900 °C and (c) 1050 °C at Ar flow rate of 100 sccm and oxygen flow rate of 10 sccm for 30 minutes	93
Figure 4.5	(a) contour and (b) 3D surface plot as a function of temperature and deposition time at fixed flow rates of Ar (60 sccm) and oxygen (30 sccm)	95
Figure 4.6	SEM images of Run 7 and Run 8 showing the effect of different temperatures with constant deposition time (60 minutes), Ar flow rate (100 sccm) and oxygen flow rate (10 sccm) with different magnifications (a) temperature of 850 °C with NWs diameter of ~61 nm (Run 7). (b) at temperature 900 °C diameter of ~70 nm (Run 8)	96
Figure 4.7	(a) Run 3 with NWs produced at temperature of 850 °C with low Ar and oxygen flow rates (b) Run 16 with NWs obtained at temperature 900 °C with high Ar and oxygen flow rates	97
Figure 4.8	Equilibrium plot with respect to temperature for reaction of Sn and O	97
Figure 4.9	Schematic diagram of deposition kinetics of the reactants involving two factors which are the temperature and pressure inside the tube (Pierson, 1992)	98
Figure 4.10	(a) Run 15 and (b) Run 16 with an average diameter of 42 nm and 248 nm respectively	99
Figure 4.11	XRD patterns of SnO <sub>2</sub> NWs grown of Si substrates for Run 15 and Run 16	100

Figure 4.12	Effect of different growth time with constant reaction temperature (850 °C) and Ar flow rate (100 sccm). (a) 30 minutes (Run 13). (b) 60 minutes (Run 15)	101
Figure 4.13	SnO <sub>2</sub> NWs growth mechanism	102
Figure 4.14	Effect of different growth time at high level temperature (900 °C) with constant oxygen and Ar flow rates (50 sccm and 100 sccm respectively). (a) 30 minutes (Run 14). (b) 60 minutes (Run 16)	103
Figure 4.15	XRD patterns of SnO <sub>2</sub> NWs grown on Si substrates for Run 14 and Run 16	104
Figure 4.16	(a) contour and (b) 3D surface plot as a function of growth time and flow rate of Ar at fixed temperature (875 °C) and oxygen flow rate (30 sccm)	105
Figure 4.17	Effect of different flow rate of Ar with constant reaction temperature (850 °C), deposition time (30 minutes) and oxygen flow rate (50 sccm). (a) 20 sccm (Run 9) and (b) 100 sccm(Run 13)flow rates	106
Figure 4.18	Contour plot of NWs's diameter as a function of temperature and flow rate of Ar at fixed deposition time (45 minutes) and oxygen flow rate (30 sccm)	107
Figure 4.19	SEM images showing the effect of the oxygen flow rate at constant temperature (900 °C), Ar flow rate (100 sccm) and deposition time (60 minutes) (a) 10 sccm flow rate of oxygen (Run 8) and (b) 50 sccm of oxygen flow rate (Run 16)	110



Figure 4.20	XRD results showing the comparison of NWs produce with varying oxygen flow rates at constant temperature, Ar flow rate and deposition time (900 °C/ 100 sccm / 60 minutes respectively)	110
Figure 4.21	EDS spectra of SnO <sub>2</sub> NWs deposited at an oxygen flow rate of (a) 10 sccm (Run 8) and (b) 50 sccm (Run 16)	111
Figure 4.22	The effect of the oxygen flow rate at low temperature (850 °C) with constant Ar flow rate (100 sccm) and deposition time (60 minutes) (a) 10 sccm flow rate of oxygen (Run 7) and (b) 50 sccm of oxygen flow rate (Run 15)	112
Figure 4.23	XRD results showing the comparison of NWs produce with varying oxygen flow rates at the highest level of temperature, Ar flow rate and deposition time (900 °C/ 100 sccm / 60 minutes respectively)	113
Figure 4.24	Graph temperature versus $\Delta G$ for reactions between Si and Au with oxygen	114
Figure 4.25	(a) contour and (b) 3D surface plot as a function of temperature and flow rate of oxygen at fixed deposition time (45 minutes) and Arflow rate (60 sccm)	115
Figure 4.26	Cube graph for different factors affecting the diameter of NWs in CVD process when (a) temperature at 850 °C (b) temperature at 900 °C	117
Figure 4.27	XRD spectra for SnO <sub>2</sub> NSs with the smallest (Run 14) and the largest crsytal size (Run 11)	119

Figure 4.28	Graph predicted versus actual value of the SnO <sub>2</sub> NSs's crystal size	122
Figure 4.29	The perturbation plot of the SnO <sub>2</sub> NSs's crystal size	124
Figure 4.30	Contribution percentage and interaction of operating parameters in response to the crystal size	124
Figure 4.31	(a) contour and (b) 3D surface plot as a function of precursor concentration and Pd concentration at fixed treatment duration (18 hour) and temperature (150 °C)	126
Figure 4.32	(a) – (d) The SEM images of Run 3 and Run 4 showing the effect of different precursor concentration with constant hydrothermal temperature (180 °C), treatment duration (12 hour) and without Pd catalyst with different magnifications, (e) XRD of both samples, (f) EDS spectra for Run 3 and (g) EDS spectra for Run 4	128
Figure 4.33	XRD spectra of Run 3 and Run 4	129
Figure 4.34	(a) – (b) The SEM images and (c) XRD pattern of Run 6 (treatment temperature of 120 °C) and Run 8 (temperature 180 °C)	133
Figure 4.35	(a) – (b) Contour and 3D surface plots showing the interaction between (a) – (b) temperature and precursor concentration, (c) temperature and treatment time and (d) temperature and Pd concentration	135

Figure 4.36	The changes in crystal size as the treatment time increase. (+ve value = the crystal size is increased with treatment time; -ve value = the crystal size is decreased with treatment; PC = Precursor concentration; Temp = temperature)	138
Figure 4.37	The SEM images of SnO <sub>2</sub> NSs at (a) 12 hour (b) 24 hour treatment time using 0.12 M of precursor concentration treated at temperature 180 °C without Pd dopant. (c) XRD patterns of both samples	139
Figure 4.38	Schematic diagram of proposed growth process of nanocubes	140
Figure 4.39	Schematic diagram of proposed growth process of NSs at different synthesis conditions	141
Figure 4.40	The interaction graph of Pd concentration and treatment time at fixed precursor concentration (0.14 M) and temperature (150 °C)	142
Figure 4.41	XRD pattern of Run 2 (0 % mol fraction Pd dopant) and Run 10 (3 % mol fraction Pd dopant)	144
Figure 4.42	(a) UV absorbance spectrums, (b) and (c) band gap calculations for SnO <sub>2</sub> NSs prepared with 0.16 M of the precursor at temperature 120 °C for 12 hour treatment duration with 0 % and 3 % Pd concentrations respectively	145
Figure 4.43	Cube graph for different factors affecting the crystallite size of SnO <sub>2</sub> NSs in hydrothermal when precursor concentration is (a) 0.12 M (b) 0.16 M	147
Figure 4.44	EDS at different spots of gas sensing element with SnO <sub>2</sub> Nanowires	151

Figure 4.45	SEM image of the gas sensing element with SnO <sub>2</sub> Nanowires after had sintered for 1 hour in 200 sccm nitrogen flow rate (a) C15 and (b) C16	151
Figure 4.46	SEM image of the gas sensing elements with SnO <sub>2</sub> NSs for(a) H3 and (b) H8	153
Figure 4.47	Graph resistance versus temperature showing the comparison of sensitivity versus temperature graph of C15 and C16	154
Figure 4.48	Graph resistance versus time of C15 upon exposure to 1000 ppm ethylene gas concentration at different temperatures	156
Figure 4.49	Real-time sensing response to ethylene gas (a) C15, (b) C16 and (c) sensitivity vs. ethylene concentration at temperature 450 °C	157
Figure 4.50	The schematic diagram of the depletion and the conduction region before and after exposure to ethylene gas	160
Figure 4.51	The illustration of the potential intra-particles connection SnO <sub>2</sub> NWs grains (Yamazaki, 2012)	162
Figure 4.52	Graph sensitivity versus temperature for hydrothermal samples with different crystal sizes	164
Figure 4.53	(a) Real time ethylene sensing characteristics and (b) the sensitivity vs. ethylene concentration of hydrothermal prepared SnO <sub>2</sub> NSs with different crystal sizes under various gas concentrations at temperature 450 °C	165

Figure 4.54	Resistance-time graphs of (a) H2 (b) H3 (c) H6 and (d) H8 at temperature 450 °C with 200 sccm flow rate of 1000 ppm ethylene gas concentration	167
Figure 4.55	SEM images of H6 (a) before sintering process and (b) after sintering process	170
Figure 4.56	SEM images of gas sensing materials after ethylene gas characterization (a) H2, (b) H3, (c) H6 and (d) H8	170
Figure 4.57	Column charts showing gas sensitivity with respect to operating temperature for (a) H3 and H11 (synthesized using 0.12 M of SnCl <sub>4</sub> .5H <sub>2</sub> O for 12 hours at temperature 180 °C) and (b) H8 and H16 (synthesized using 0.16 M of SnCl <sub>4</sub> .5H <sub>2</sub> O for 24 hours at temperature 180 °C)	174
Figure 4.58	Graph resistance versus time of H16 upon exposure to 1000 ppm ethylene gas concentration at different temperatures	175
Figure 4.59	Real time ethylene sensing characteristics of (a) H11 at temperature 350 °C (b) H16 at temperature 375 °C	176
Figure 4.60	Resistance in air (Ra) value in 1000 ppm ethylene gas concentration at temperature 350 °C	179
Figure 4.61	Gas sensing setup at Hanoi University of Science and Technology (HUST), Hanoi, Vietnam (Wan et al., 2013)	184

## LIST OF ABBREVIATIONS

Ar	Argon
Sn	Tin or Stannum
SnO <sub>2</sub>	Tin oxide
SnSO <sub>4</sub>	Tin (II) sulfate
Pd	Palladium
Pt	Platinum
PdCl <sub>2</sub>	Palladium (II) chloride
NaOH	Sodium hydroxide
NWs	Nanowires
NSs	Nanostructures
SEM	Scanning electron microscopy
EDS	Energy dispersive spectroscopy
XRD	X-ray diffraction spectroscopy
UV-Vis	Ultra-violet visible spectroscopy
DOE	Design of Experiment
ANOVA	Analysis of variance
CV	Coefficient of variance
ppm	Part per million
ppb	Part per billion
VLS	Vapor-liquid-solid

## LIST OF SYMBOLS

$^{\circ}\text{C}$	Degree Celsius
$\leq$	Less than or equal to
$\Omega$	Ohm
$\Delta G$	Gibbs free energy
$\%$	Percentage
$\text{\AA}$	Angstrom
$\lambda$	Wavelength of Cu-K $\alpha$ line
$\beta$	Full width at half maximum
$\theta$	Bragg angle
$K$	Shape factor
$\varphi_m$	Schottky barrier
$\mu\text{m}$	Micro-meter
$\text{nm}$	Nano-meter
$s$	Second

# **PENGHASILAN BAHAN PENGESAN GAS BERSKALA NANO SEBAGAI PENGESAN GAS ETILENA**

## **ABSTRAK**

Gas etilena adalah bahan penting dalam pemasaran produk pertanian segar kerana ia boleh digunakan secara komersial untuk hormon tanaman tiruan, mengawal dan memantau proses pemasakan buah-buahan klimaterik. Menyedari kepentingan alat pengesan gas etilena untuk proses pemasakan buah-buahan, banyak kajian telah dijalankan untuk mengkaji pengaruh gas etilena dalam proses pemasakan tanaman. Nanopartikel tin oksida ( $\text{SnO}_2$ ) adalah bahan yang paling popular untuk mengesan gas etilena kerana ia adalah semikonduktor jenis n yang mempunyai jalur jurang yang luas, justeru itu, menyebabkan rintangan elektrik yang rendah dan pengkonduksian elektrik yang lebih baik untuk alat pengesan gas. Tambahan pula, suhu operasi yang rendah, kepekaan yang tinggi, reka bentuk sensor yang ringkas serta kos pembuatan yang rendah membuat  $\text{SnO}_2$  pilihan terbaik untuk aplikasi pengesan gas. Dalam penyelidikan ini, pemendapan wap kimia (CVD) dan kaedah hidroterma telah digunakan untuk mensintesis nano-struktur  $\text{SnO}_2$  ( $\text{SnO}_2$  NSs). Sintesis, pencirian bahan dan sifat-sifat pengesan gas etilena telah dikaji dengan menggunakan nano  $\text{SnO}_2$ . Pertama sekali, nano-wayar (NWs)  $\text{SnO}_2$  telah disintesis di atas substrat silikon dengan menggunakan kaedah CVD. Kesan daripada pemanipulasian pembolehubah CVD (seperti suhu sintesis, tempoh sintesis, kadar aliran gas argon dan gas oksigen) terhadap dimensi  $\text{SnO}_2$  NWs telah disiasat dengan menggunakan analisa statistik iaitu Reka Bentuk Eksperimen (RBE) oleh perisian Design Expert 6.0.8. Mikroskop imbasan elektron (SEM), spektroskop serakan tenaga (EDS) dan spektroskop pembelauan sinar-X (XRD) telah mengesahkan fabrikasi  $\text{SnO}_2$  NWs.